

CONTINUATION-IN-PART

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## A COMPRESSED FLUID FORMULATION

MAIL STOP PATENT APPLICATION

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## **A COMPRESSED FLUID FORMULATION**

### **CROSS-REFERENCE TO RELATED APPLICATIONS**

This is a continuation-in-part of application Serial No. 10/033,458,  
5 filed December 27, 2001 entitled "A Compressed Fluid Formulation."

Reference is made to commonly assigned, copending application  
Serial No. 10/033,422 (DN 83878), entitled A COMPRESSED FLUID  
FORMULATION CONTAINING ELECTROLUMINESCENT MATERIAL,  
filed December 27, 2001. The copending application is incorporated by reference  
10 herein for all that it contains.

### **FIELD OF THE INVENTION**

This invention relates generally to imaging compositions that  
contain functional materials that are dispersed and/or solubilized in a fluid that is  
15 in a compressed state. The compositions are used to create a high-resolution  
pattern or image onto a substrate.

### **BACKGROUND OF THE INVENTION**

Color filters are produced via numerous technologies including  
20 vapor deposition, spin-coating, laser-thermal deposition, mordant capturing  
receivers and a host of other methods. Often, the color filters are formed by at  
least one of the following procedures for example forming the color filter using a  
single color in the form of a contiguous film or an array of pixels, combinations of  
red, green, and blue or cyan, magenta, yellow, and black pixels in a two  
25 dimensional array to allow for multicolor applications. The color filter forming  
materials are composed of organic and organometallic pigments, semiconductors,  
ceramics and combinations thereof.

The color filter forming materials are typically provided by one of  
three methods. One method is by wet formulation requiring a solvent, dispersants,  
30 at least one filter forming material, and other formulation additives. A second  
method uses a dry, flexible donor sheet of at least one filter forming material in a

thin film that has been coated on a flexible substrate to be used in thermal deposition. The third method provides the color filter forming material as a dry powder in a source boat container for vapor deposition purposes. In most instances, the dry powder is placed one powder per source boat.

5 Ink jet recording or printing systems are commonly used to create high-resolution patterns on a substrate .In a typical ink jet recording or printing system, ink droplets are ejected from a nozzle towards a recording element or medium to produce an image on the medium. The ink droplets, or recording liquid, generally comprise a functional material or functional material, such as a  
10 dye or pigment or polymer, and a large amount of solvent. In conventional inkjet printing systems, the liquid ink droplets are ejected from the nozzle using pressure pulses generated by an oscillating piezoelectric crystal or by heating the nozzle to generate an ink droplet resulting from bubble formation or from ink phase change. The solvent, or carrier liquid, typically is made up of water, an organic material  
15 such as a monohydric alcohol, a polyhydric alcohol or mixtures thereof. There can be many additives in the system aimed at preserving the pixel integrity upon deposition to the receiver. Such materials may be surfactants, humectants, biocides, rheology modifiers, sequestrants, pH adjusters, and penetrants among others. Such materials are necessary due to the high water loads in conventional  
20 ink formulations. More recently, the inkjet printing method has been used to make color filter display devices.

U.S. Pat. No. 6,245,393 discloses a method of making a multicolor display device, comprising a transparent substrate, fluorescent dye deposited in a dye layer on to a substrate by inkjet printing. There is a problem with this  
25 invention in that the inkjet printing compositions, which contain fluorescent dyes, have high solvent loads to be used in conventional ink jet printers. It is therefore necessary to have processing steps and time devoted to removal of the solvent materials. There are alternate technologies that are available in prior art, that eliminate this problem by using gaseous propellants.. For example, Peeters et al.,  
30 in U.S. Pat. No. 6,116,718, disclose a print head for use in a marking apparatus in which a propellant gas is passed through a channel, the functional material is

introduced controllably into the propellant stream to form a ballistic aerosol for propelling non-colloidal, solid or semi-solid particulate or a liquid, toward a receiver with sufficient kinetic energy to fuse the marking material to the receiver.

There is a problem with this technology in that the functional material and

5 propellant stream are two different entities and the propellant is used to impart kinetic energy to the functional material. This can cause functional material agglomeration leading to nozzle obstruction and poor control over functional material deposition. Another problem with this technology is that when the functional material is added into the propellant stream in the channel it forms a

10 non-colloidal ballistic aerosol prior to exiting the print head. This non-colloidal ballistic aerosol, which is a combination of the functional material and the propellant, is not thermodynamically stable. As such, the functional material is prone to settling in the propellant stream, which in turn, can cause functional material agglomeration leading to nozzle obstruction and poor control over

15 functional material deposition.

Thermal dye transfer printers use a transfer ribbon made of a plastic film. Page-sized panels on the ribbon consist of cyan, magenta and yellow dye in a solid form. A thermal print head, consisting of thousands of heating elements, capable of precise temperature variations, moves across the transfer ribbon. Heat

20 from the heating elements cause the color on the ribbon to vaporize and diffuse onto the surface of the specially coated paper. Precise temperature variations are responsible for the varying densities of color. The hotter the heating element, the more dye is vaporized and diffused onto the paper's surface. Problems with this technology include the functional material, which is solid and is converted into

25 gas, then deposited on the surface of the receiver. Also, thermally unstable materials cannot be used in the donor sheet. The receiver must be specially designed for the dye sublimation printing and include materials, which may require an overcoat for protection. This process requires a separate step in that the formulation needs to be coated separately via a curtain-coating process on to a

30 transfer ribbon or substrate, which is then used in printing. U.S. Pat.

No. 5,874,188 discloses a method of making a color filter array comprising organic

pigment material that is deposited via thermal transfer onto a patterned substrate wherein the pattern is developed by lithographic techniques. There is a problem with this invention in that the organic pigment material that is transferred thermally must be previously coated onto a thermal transfer donor sheet requiring 5 multiple processing steps, time, and additional costs. Further, the organic pigments typically are coated out of solvent-based systems requiring the use of high solvent loads and subsequent removal and disposal of the often toxic solvents. Further disadvantages of the present invention are the multiple lithographic masking steps that are required to deposit the different colors of 10 organic pigment, which are complex, tedious, and time consuming

Technologies that use supercritical fluid solvents to create thin films are also known. For example, R.D. Smith in U.S. Patent 4,734,227, issued March 29, 1988, discloses a method of depositing solid films or creating fine powders through the dissolution of a solid material into a supercritical fluid 15 solution and then rapidly expanding the solution to create particles of the functional material in the form of fine powders or long thin fibers which may be used to make films. There is a problem with this method in that the free-jet expansion of the supercritical fluid solution results in a non-collimated/defocused spray that cannot be used to create high-resolution patterns on a receiver. Further, 20 defocusing leads to losses of the functional material. There is yet another problem with the Smith method in that very few materials can be completely dissolved in supercritical fluid solutions, and this restricts the use of many common materials used in various applications, which are not soluble in supercritical fluids.

Huck et al., in WO 02/45868 A2, discloses a method of creating a 25 pattern on a surface of a wafer using compressed carbon dioxide. The method includes dissolving or suspending a material in a solvent phase containing compressed carbon dioxide, and depositing the solution or suspension onto the surface of the wafer, the evaporation of the solvent phase leaving a patterned deposit of the material. The wafer is prepatterned using lithography to provide the 30 wafer with hydrophilic and hydrophobic areas. After deposition of the solution (or suspension) onto the wafer surface followed by the evaporation of the solvent

phase, the material (a polymer) adheres to one of the hydrophobic and hydrophilic areas. The solution (or suspension) is deposited on the wafer surface either in the form of liquid drops or a feathered spray.

This method is disadvantaged because deposition using a feathered spray  
5 requires that the wafer surface be prepatterned prior to deposition. Hence, direct patterning of the wafer surface is not possible because of the diverging profile (feathered) of the spray. Additionally, a wafer surface that has not been prepatterned cannot be patterned using this method. This method also requires time for drying so that the solvent phase of the liquid drops (or feathered spray)  
10 can evaporate. During the time associated with solvent phase evaporation, the solvent and the material can diffuse from one pixel to next(for example, into the surface or along the surface) degrading the desired pattern.

A different approach for marking is needed—one that would eliminate the need for the "water management" additives. There is also a need for  
15 a technology that permits high speed, accurate, and precise deposition of a functional material on a receiver. There is also a need for a technology that permits functional material deposition of ultra-small (nano-scale) particles. There is also a need for a technology that permits high speed, accurate, and precise patterning of a receiver that can be used to create high-resolution patterns on a  
20 receiver. There is also a need for a technology that permits high speed, accurate, and precise patterning of a receiver having reduced material agglomeration characteristics.

### SUMMARY OF THE INVENTION

The present invention overcomes the problems discussed above by  
25 providing an imaging composition comprising a mixture of a fluid and a functional material. The fluid is compressed and the functional material is dispersed and/or solubilized in the compressed fluid. The mixture is thermodynamically stable or thermodynamically metastable or both.

The present invention also provides an imaging composition  
30 comprising a mixture of a carbon dioxide and a colorant. The carbon dioxide is compressed and the colorant is dispersed and/or solubilized in the compressed

carbon dioxide. The mixture is thermodynamically stable or thermodynamically metastable or both. The invention is useful for inkjet, organic light emitting diode display, color filter arrays, coating applications, polymer filler, and thin film formation applications.

5           The present invention discloses:

An imaging composition comprising a mixture of a fluid, functional material, a surfactant and/or a dispersant;

              wherein the fluid is compressed and the functional material is dispersed and/or solubilized in the compressed fluid; and

10           wherein the mixture is thermodynamically stable or thermodynamically metastable or both.

The present invention also discloses:

An imaging composition comprising a mixture of a fluid , a color filter material, a surfactant and/or a dispersant ;

15           wherein the fluid is compressed and the color filter material is dispersed and/or solubilized in the compressed fluid; and

              wherein the mixture is thermodynamically stable or thermodynamically metastable or both.

20

## BRIEF DESCRIPTION OF THE DRAWINGS

In the detailed description of the preferred embodiments of the invention presented below, reference is made to the accompanying drawings.

25           Figure 1 is a photomicrograph of lines drawn using compressed fluid formulation containing Duasyn Acid blue AE-02 ® dye used in the present invention. The dashed lines are produced with widths of approximately 100-200 microns.

30           Figure 2 is a photomicrograph of lines drawn using compressed fluid formulation containing Duasyn Acid blue AE-02 ® dye used in the present invention. The dashed lines are produced with lengths approximately 1.5-2 millimeters.

Figure 3 is a photomicrograph of dots drawn using compressed fluid formulation containing Duasyn Acid blue AE-02 ® dye used in the present invention. The dots are produced with widths of approximately 2 millimeters.

5

## DETAILED DESCRIPTION OF THE INVENTION

The formulations useful in the present invention contain a functional material, which is dispersed and / or solubilized, in a compressed fluid. The compressed fluid is any material with a density greater than 0.1 grams/cc. The compressed fluid may include a compressed liquid and /or a supercritical fluid. Materials that are at sufficiently high temperatures and pressures below their critical point are known as compressed liquids. Materials in their supercritical fluid and/or compressed liquid state that exist as gases at ambient conditions find application here because of their unique ability to solubilize and/or disperse functional materials of interest in the compressed liquid or supercritical state. In this context, the chosen materials taken to a compressed liquid and/or supercritical fluid state are gases at ambient pressure and temperature. Ambient conditions are preferably defined as temperature in the range from -100 to +100 °C, and pressure in the range from  $1 \times 10^{-8}$  - 100 atm for this application. More commonly, the ambient conditions are temperature in the range of 0 to 100°C and pressure in the range from  $1 \times 10^{-5}$  to 100 atm. for this application. One skilled in the art should know how to select and maintain the appropriate ambient conditions such that the selected compressed fluid is gas at the ambient conditions.

The compressed fluids include, but are not limited to, carbon dioxide, nitrous oxide, ammonia, xenon, ethane, ethylene, propane, propylene, butane, isobutane, chlorotrifluoromethane, monofluoromethane, sulphur hexafluoride and mixtures thereof. Due its characteristics, e.g. low cost, wide availability, etc., carbon dioxide is generally preferred in many applications.

Functional materials can be any material that needs to be delivered to a receiver, for example imaging dyes, ceramic nanoparticles etc., to create a pattern on the receiver by deposition, etching, coating, other processes involving the placement of a functional material on a receiver, etc.

The functional materials may be selected from species that are ionic and/or molecular of the types such as organic, inorganic, metallo-organic, polymeric, oligomeric, metallic, alloy, ceramic, a synthetic and/or natural polymer, and a composite material of these previously mentioned. The functional material  
5 can be a solid or a liquid. Additionally, the functional material can be an organic molecule, a polymer molecule, a metallo-organic molecule, an inorganic molecule, an organic nanoparticle, a polymer nanoparticle, a metallo-organic nanoparticle, an inorganic nanoparticle, an organic microparticles, a polymer micro-particle, a metallo-organic microparticle, an inorganic microparticle, and/or composites of  
10 these materials, etc. After suitable mixing with the compressed fluid the functional material is uniformly distributed within a thermodynamically stable/metastable mixture, that can be a dispersion, with the compressed fluid.

The functional materials, more specifically, color filter materials may be selected from species that are ionic and/or molecular of the types such as 15 organic, inorganic, metallo-organic, polymeric, oligomeric, metallic, alloy, ceramic, a synthetic and/or natural polymer, and a composite material of these previously mentioned. For example, color filter material may be Copper Phthalocyanine, Formula IV, Disperse Red 60 (1-amino-4-hydroxy-2-phenoxy-9,10-anthraquinone), Formula V, or Coumarin-545T (C545T), Formula VI.

20 Additionally, color filter materials which are useful in the invention include the following: phthalocyanines, such as Pigment Blue 15, nickel phthalocyanine, chloroaluminum phthalocyanine, hydroxy aluminum phthalocyanine, vanadyl phthalocyanine, titanyl phthalocyanine, and titanyl tetrafluorophthalocyanine; isoindolinones, such as Pigment Yellow 110 and  
25 Pigment Yellow 173; isoindolines, such as Pigment Yellow 139 and Pigment Yellow 185; benzimidazolones, such as Pigment Yellow 151, Pigment Yellow 154, Pigment Yellow 175, Pigment Yellow 194, Pigment Orange 36, Pigment Orange 62; Pigment Red 175, and Pigment Red 208; quinophthalones, such as Pigment Yellow 138; quinacridones, such as Pigment Red 122, Pigment Red 202,  
30 and Pigment Violet 19; perylenes, such as Pigment Red 123, Pigment Red 149, Pigment 179, Pigment Red 224, and Pigment Violet 29; dioxazines, such as

- Pigment Violet 23; thioindigos, such as Pigment Red 88, and Pigment Violet 38; epindolidiones, such as 2,8-difluoroepindolidione; anthanthrones, such as Pigment Red 168; isoviolanthrones, such as isoviolanthrone; indanthrones, such as Pigment Blue 60; imidazobenzimidazolones, such as Pigment Yellow 192;
- 5 pyrazoloquinazolones, such as Pigment Orange 67; iketopyrrolopyrroles, such as Pigment Red 254, Irgazin DPP RubinTR, Cromophthal DPP OrangeTR; Chromophthal DPP Flame Red FP (all of Ciba-Geigy); and bisaminoanthrones, such as Pigment Red 177.

Additionally, the functional materials can be functionalized to  
10 dissolve, disperse and/or solubilize the functional material in the compressed fluid. The functionalization may be performed by attaching fluorocarbons, siloxane, hydrocarbon functional groups to the electroluminescent material.

Additionally, the formulation may contain a dispersant and or a surfactant to solubilize and/or disperse the functional material. The dispersant  
15 and/or surfactant can be selected from any group that will have appropriate solubility in the compressed liquid and/or supercritical fluid medium as well as have interactions with the functional material so that the functional material can be solubilized. Such materials include, but are not limited to, fluorinated polymers such as perfluoropolyether, siloxane compounds, etc. The surfactants preferred in  
20 the invention include Fluorolink 7004® (Ausimont Montedison Group) and Fomblin MF-300 ® (Ausimont Montedison Group).

Additionally, the ratio of surfactant to functional material in the formulation is from about 0.1:1 to about 500:1. More preferably, the ratio of surfactant to functional material is from about 1:1 to about 100:1. In yet another  
25 preferred embodiment of the invention, the ratio of co-solvent to functional material in the formulation is from about 0.01:1 to about 100:1. In still another embodiment of the invention, the ratio of compressed fluid to functional material in the formulation is from about 1:1x10<sup>5</sup> to about 1:20.

The compressed fluid forms a continuous phase and the functional  
30 material dispersed and/or solubilized in the compressed fluid forms a single phase. The formulation is maintained at a temperature and a pressure suitable for the

functional material and the compressed fluid used in a particular application. More commonly, the formulation conditions are temperature in the range of 0 to 100°C and pressure in the range from  $1 \times 10^{-2}$  to 400 atm. for this application.

The method of preparing the formulation will now be discussed.

- 5      The apparatus used for making the formulation has been disclosed in the pending U.S. application Serial No. 09/794,671, which is incorporated here in its entirety. Briefly, the functional material is controllably introduced into the formulation reservoir. The compressed fluid is also controllably introduced into the formulation reservoir. The contents of the formulation reservoir are suitably
- 10     mixed using mixing device to ensure intimate contact between the functional material and compressed fluid. As the mixing process proceeds, functional material is solubilized or dispersed within the compressed fluid. The process of dissolution/dispersion, including the amount of functional material and the rate at which the mixing proceeds, depends upon the functional material itself, the
- 15     particle size and particle size distribution of the functional material (if the functional material is a solid), the compressed fluid used, the temperature, and the pressure within the formulation reservoir. When the mixing process is complete, the mixture or formulation of functional material and compressed fluid is thermodynamically stable/metastable in that the functional material is dissolved or
- 20     dispersed within the compressed fluid in such a fashion as to be indefinitely contained in the same state as long as the temperature and pressure within the formulation chamber are maintained constant. This state is distinguished from other physical mixtures in that there is no settling, precipitation, and/or agglomeration of functional material particles within the formulation chamber
- 25     unless the thermodynamic conditions of temperature and pressure within the reservoir are changed. As such, the functional material and compressed fluid mixtures or formulations of the present invention are said to be thermodynamically stable/metastable.

- The method for delivering the formulation to a suitable receiver
- 30     will now be discussed. The apparatus used for delivering the formulation to a suitable receiver has been disclosed in the pending U.S. application Serial No.

09/794,671; which is incorporated here in its entirety. Briefly, the functional material is precipitated from the compressed fluid by manipulating and/or changing the temperature and/or pressure conditions. The precipitated functional material is directed towards the receiver as a suitable shaped stream. The  
5 compressed fluid containing the functional material will be expanded through a suitable orifice into an ambient atmosphere where the compressed fluid will become a gas. The functional material will begin to precipitate non-reactively into particles and/or agglomerates of particles with the dispersant and/or surfactant coating the surfaces of these particles and/or agglomerates thereby limiting the  
10 growth of particles. The precipitated and/or aggregated functional material, free of compressed fluid, is deposited on a receiver in a precise and accurate fashion to form a desired image. Hence the receiver is instantaneously dry upon delivery of the functional material on the receiver.

The receiver can be any solid including an organic, an inorganic, a  
15 metallo-organic, a metallic, an alloy, a ceramic, a synthetic and/or natural polymeric, a gel, a glass, and a composite material. The receiver can be porous or non-porous.

The size of the precipitated nanomaterials can be controlled by the ratio of functional material to dispersant and/or surfactant. The size of the  
20 precipitated nanomaterials can be controlled by the depressurization step through suitable orifice design and optimization with temperature of solution, pressure of solution, flow rate of solution, and concentrations of the functional materials and dispersant and/or surfactants. The size of the precipitated nanomaterials can be controlled by the appropriate selection of dispersant and/or surfactant material  
25 such as the type of functional groups on the molecule as well as the solubility in the particular compressed liquid and/or supercritical fluid. Typical particle size of the functional material deposited on the receiver is in the range of 1 nanometer to 1000 nanometers. More preferably, the particle size of the functional material is in the range of 1 nanometer to 100 nanometers.

30 The precipitated nanomaterial can also be collected by any number of methods. For example, the precipitated nanomaterials may be injected

into/onto a suitable substrate sheet for immobilization or the nanomaterials may be collected in a suitable liquid. Due to the surfactant coating of the nanomaterials during the depressurization process, the nanomaterials will be stable and not undergo significant agglomeration. Thereby, discrete nanoparticles can be obtained depending on the processing conditions.

It is to be understood that elements not specifically shown or described may take various forms well known to those skilled in the art. Additionally, materials identified as suitable for various facets of the invention, for example, functional materials. These are to be treated as exemplary, and are not intended to limit the scope of the invention in any manner.

## EXAMPLES

### **Example 1 Preparation of a formulation containing Duasyn Acid Blue AE-02**

#### **® dye, a colorant and a color filter material**

0.01 g of Duasyn Acid Blue AE-02 ® (Clariant Corp.) and 0.649 g of Fomblin MF-300 ® (Ausimont Montedison Group) (Formula II) and 6.82 g of CO<sub>2</sub> (Matheson Group) were placed in a high-pressure cell at 23 °C and the pressure was adjusted to 204 atm (3000 psig). Visual examination of the view cell suggested that the formulation in the system was a homogeneous, single phase.

This was further confirmed when the cloud point of the system was determined to be at 86 atm.(1258 psig).

### **Example 2: Preparation of another formulation containing Duasyn Acid Blue AE-02 ® dye with a different surfactant**

0.01 g of Duasyn Acid Blue AE-02 ® (Formula III) and 0.649 g of Fluorolink 7004 ® (Ausimont Montedison Group) and 6.82 g of CO<sub>2</sub> (Matheson Group) were placed in a high pressure cell at 40 °C and the pressure was adjusted to 150 atm. Visual examination of the view cell suggested that the formulation in the system was a homogeneous, single phase.

### **Example 3: Preparation of another formulation containing Copper Pthalocyanine, an Inkjet Functional Material and a color filter material**

0.0126 g of Copper Phtalocyanine (Formula V), 0.4763 g of Fluorolink ® 7004, and 7.06 g of CO<sub>2</sub> were placed in a high pressure cell at 25.3°C and at 150 atm and mixed. After an appropriate time, the system was visibly homogeneous. The formulation was expanded to ambient condition through a needle valve for 5 seconds.

**Example 4: Writing a line using the formulation prepared in Example 2 (with lower frequency actuation)**

The formulation from Example 2 was kept at 150 atm and 40 °C in the formulation reservoir. This formulation is expanded through a nozzle, with a 300 micron throat. The nozzle was actuated at 30 Hz. The distance between the exit of the nozzle and the substrate is set at a gap of 500 micron. A substrate translation speed of 2 inches/ second was used. Figure 1 shows the resulting dashed lines produced with widths of approximately 100-200 microns. Figure 2 shows the resulting dashed lines produced with lengths of approximately 1.5-2 millimeters.

**Example 5: Imaging dots using the formulation prepared in Example 2 (with higher frequency actuation)**

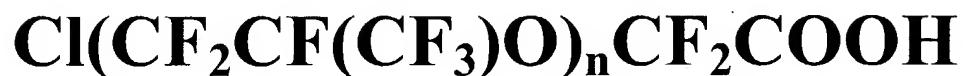
The formulation from Example 2 was kept at 150 atm and 40 °C in the formulation reservoir. This formulation is expanded through a nozzle, with a 300 micron throat. The nozzle was actuated at 150 Hz. The distance between the exit of the nozzle and the substrate is set at a gap of 500 micron. A substrate translation speed of 2 inches/ second was used. Figure 3 shows the resulting dots produced with widths of approximately 2 millimeters.

**Example CF1: Preparation of formulation containing C-545T, a color filter material**

0.0058 g of C-545T (Formula V), 0.555 g of ammonium exchanged Fluorolink 7004 ® (Ausimont Montedison Group), and 5.0 g of CO<sub>2</sub> were placed in a high pressure cell at 25.3°C and 150 atm. and mixed. After an appropriate time, the system was visibly homogeneous. The formulation was expanded to ambient condition through a needle valve for 5 seconds to deposit C-545T on a substrate.

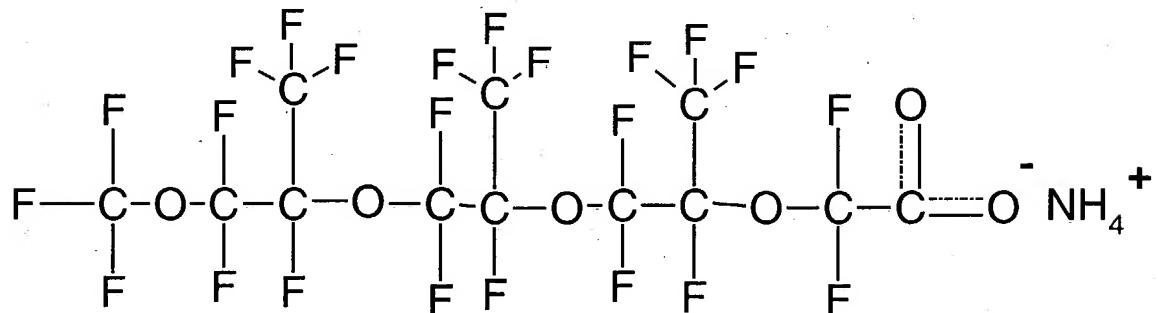
**Formula I:** Chemical structure of surfactant Fluorolink 7004® used in the present invention

5



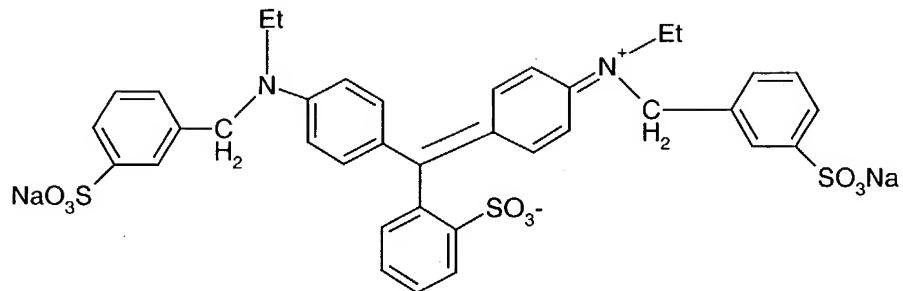
10

**Formula II:** Chemical structure of surfactant Fomblin MF-300 ® used in the present invention



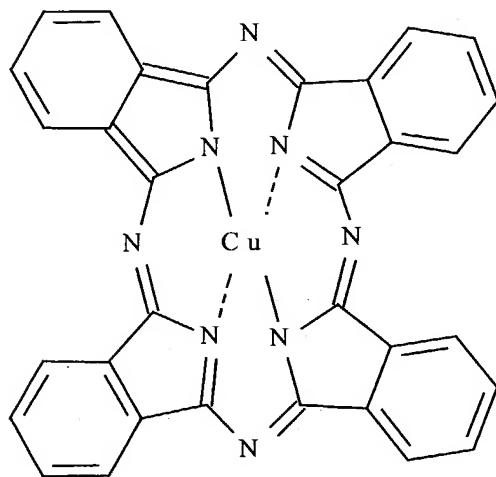
**Formula III:** Chemical structure of Duasyn Acid blue AE-02 ® dye used in the present invention

5

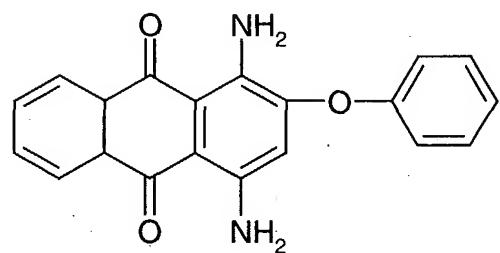


**Duasyn Acid Blue AE-02  
(Acid Blue 9)  
MW ~ 793 g/mol**

10    **Formula IV:** Chemical structure of copper pthalocyanine used in the present invention



**Formula V:** Chemical structure of Disperse Red 60 (1-amino-4-hydroxy-2-phenoxy-9,10-anthraquinone), used in the present invention



**Formula VI:** Chemical structure of Coumarin-545T (C545T), used in the present invention

